Varying processing parameters in the development of slurry-based oxide bond coat for environmental barrier coatings

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Introduction

- Silicon carbide (SiC)-based ceramic matrix composites (CMCs) are an attractive alternative to nickel-base superalloys as hot-section aircraft engine components
  - Lower density and higher operating temperature improve engine efficiency
- CMCs require an environmental barrier coating (EBC) to prevent volatilization of the protective SiO_2 scale formed on SiC in a combustion environment

Oxidation

$$SiC + \frac{3}{2}O_2(g) \rightarrow SiO_2 + CO(g)$$

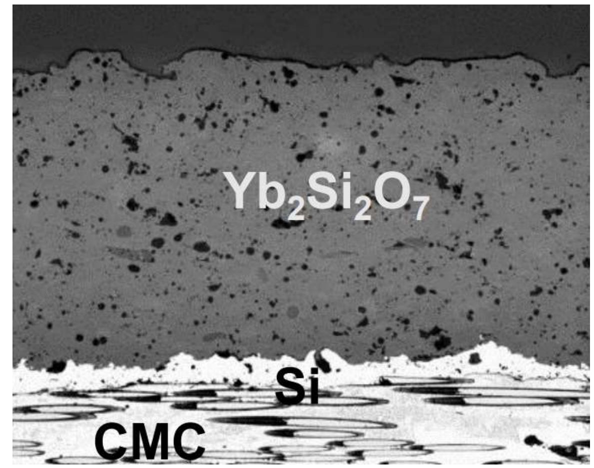
$$SiC + 3H_2O(g) \rightarrow SiO_2 + CO(g) + 3H_2(g)$$

Recession

$$SiO_2 + 2H_2O(g) \rightarrow Si(OH)_4(g)$$

Motivation

- Current-generation EBCs consist of a rare earth (RE) silicate ($RE_2Si_2O_7$; RE_2SiO_5) top coat and a Si bond coat
- Upper use temperature limited by melting point of Si bond coat (~1410°C)
  - Operating temperature of 2700°F (1482°C) desired



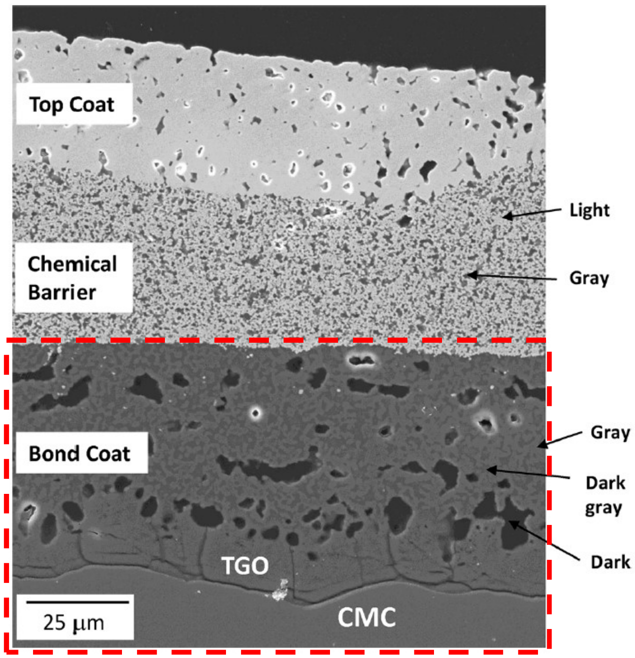
https://ntrs.nasa.gov/api/citations/20180004253

Motivation

- A slurry-based oxide bond coat with significantly higher temperature capabilities has been developed at NASA Glenn Research Center
  - Three-layer system
  - Cycling at 1427°C in 90 vol% H_2O/10 vol% O_2

	Mullite	$Yb_2Si_2O_7$	$HfSiO_4$	Al_2O_3	Si
Top coat	0.2 – 1 wt%	balance			0 – 10 wt%
Chemical barrier			balance		10 wt%
Bond coat	balance	1.3 wt%		7	20 wt%

500 h at 1427°C; ~15 µm thick TGO



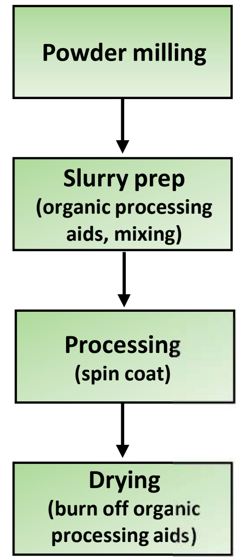
KN Lee et al., J. Eur. Ceram. Soc., 41 1639–1653 (2021).

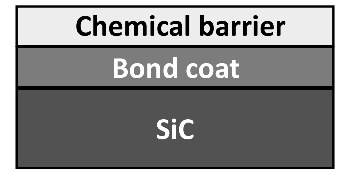
Objective

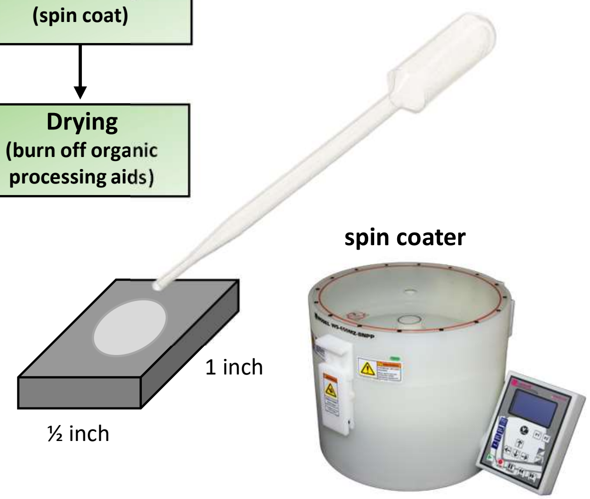
- Explore the effects of processing and chemistry on slurry-based oxide bond coat performance at ~1480°C in a steam cycling environment

Experimental

- Slurry processes (dip, spray, spin) provide a feasible, inexpensive, and scalable method for producing coatings
- Slurry = powder + solution (solvent + binder + dispersant)
- A bond coat (5 mil) and chemical barrier (5 mil) were deposited on Hexoloy® SA coupons via spin coating





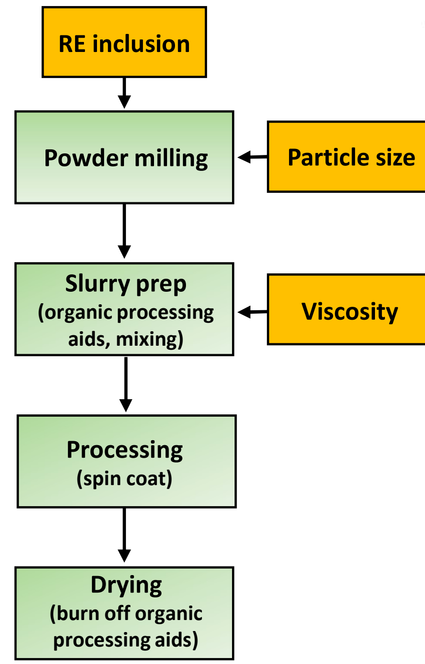


	Mullite	$RE_2Si_2O_7$	$HfSiO_4$	Al_2O_3	Si	SiC
Chemical barrier			balance		2 wt%	
Bond coat	balance	≤ 1 wt%		< 1 wt%	15 wt%	5 wt%

KN Lee, DL Waters, U.S. Patent 11325869, 10 May 2022.

Experimental

- Bond coat variables:
  - Particle size
    - Powders prepared with 2, 3, or 5 mm diameter ZrO_2 milling media
  - Viscosity
    - Slurry viscosity varied between ~10 (“low viscosity”) and 20 – 30 cP (“standard viscosity”)
  - RE inclusion
    - Yb/Sc/Lu



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Experimental

- Dried coupons sintered for 3 h at ~1525°C and annealed for 10 h at ~1480°C in a stagnant-air box furnace
- Sintered/annealed samples exposed in a steam cycling rig (1 hour hot, 30 min cool) at ~1480°C
  - 90 vol% H_2O/10 vol% O_2
  - 10 cm/s gas velocity
- Coating performance evaluated macroscopically and by scanning electron microscopy (SEM)

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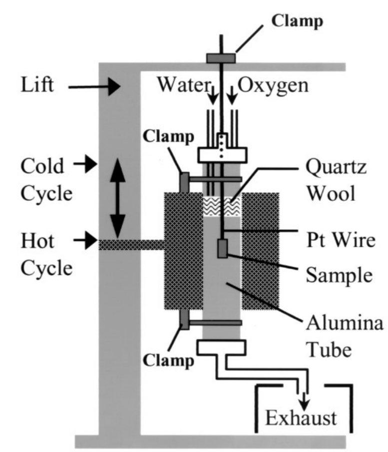
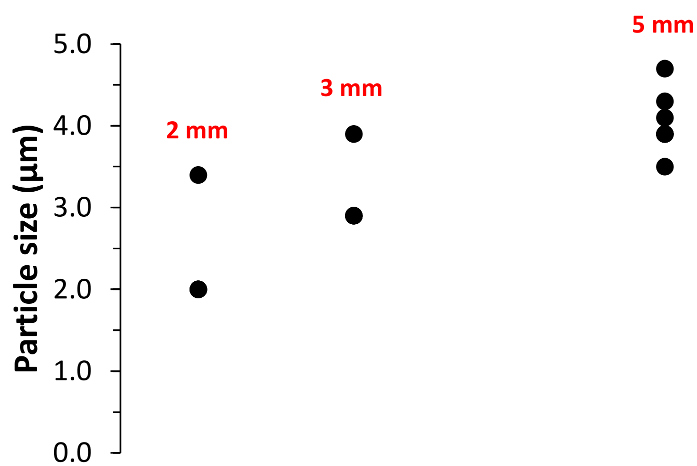
Powder milling
↓
Slurry prep (organic processing aids, mixing)
↓
Processing (spin coat)
↓
Drying (burn off organic processing aids)
↓
Sintering and annealing
→
Testing and Characterization

KN Lee et al., J. Am. Ceram. Soc., 86 [8] 1299–1306 (2003).



Results: Particle size

- Average particle size ranged from 2.0 to 4.7 µm
- Smaller particle size = denser bond coat?
  - ↑ density, ↑ elastic modulus, ↑ thermal stress during cycling



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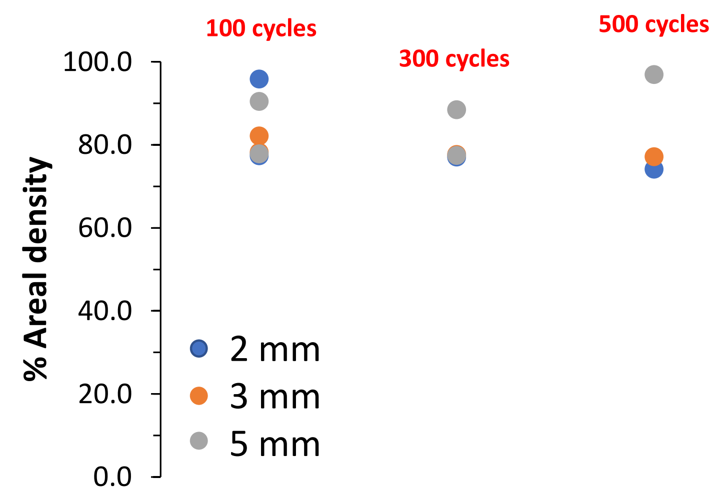

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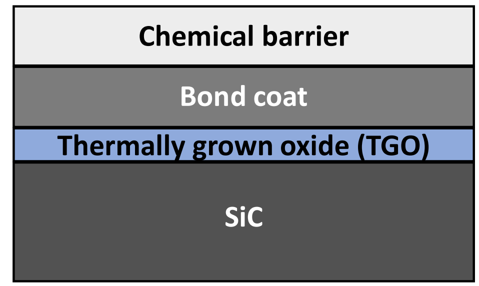
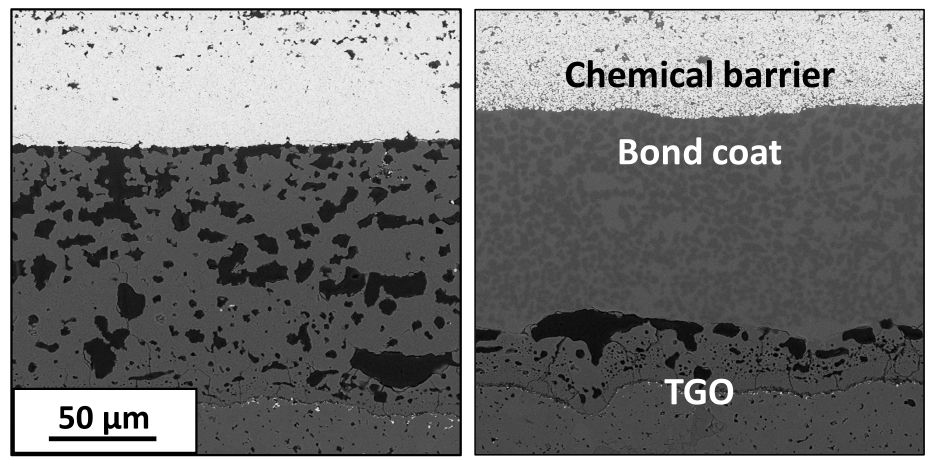


Particle size = 2.0 µm; 500 cycles, ~1480°C



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No apparent trends with respect to density or performance as a function of particle size.

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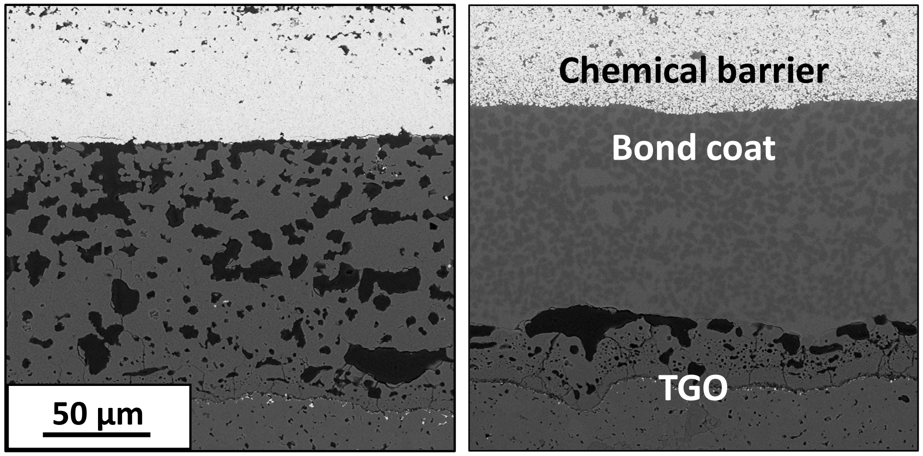

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- “Standard” viscosity = powder to solution ratio of 36/64 (wt%)
- “Low viscosity” = powder to solution ratio of 25/75 (wt%)

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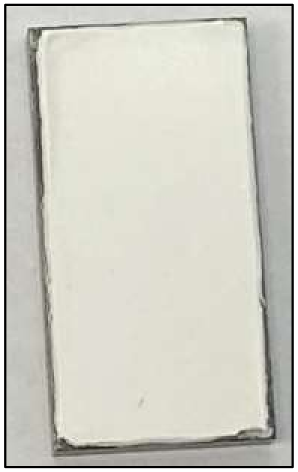
- Low viscosity samples generally showed improved resistance to edge-lifting/edge effects after processing

Standard viscosity

Low viscosity

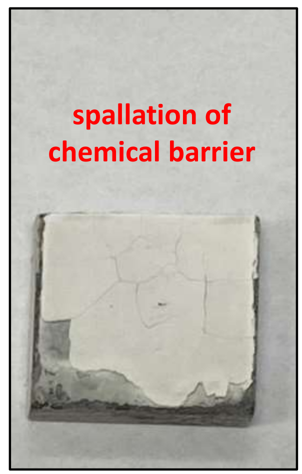


As-processed

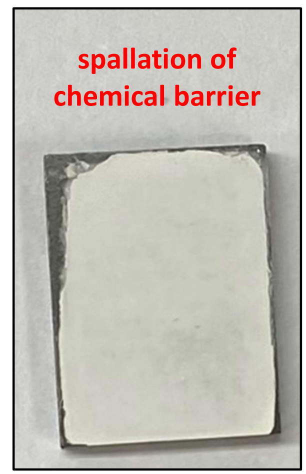
Results: Viscosity

- Low viscosity samples generally showed improved resistance to edge-lifting/edge effects after processing
- Only minor improvements in bond coat performance in a steam cycling environment were observed across multiple samples

Standard viscosity



Low viscosity



After 500 cycles, ~1480°C

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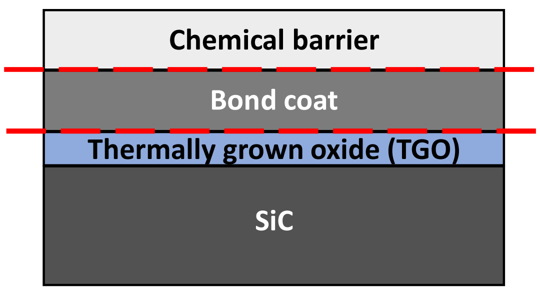

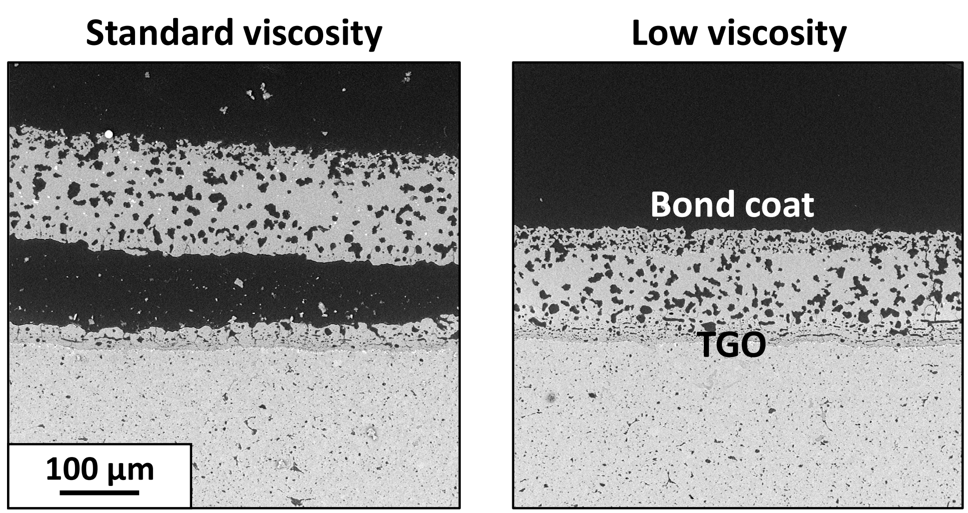

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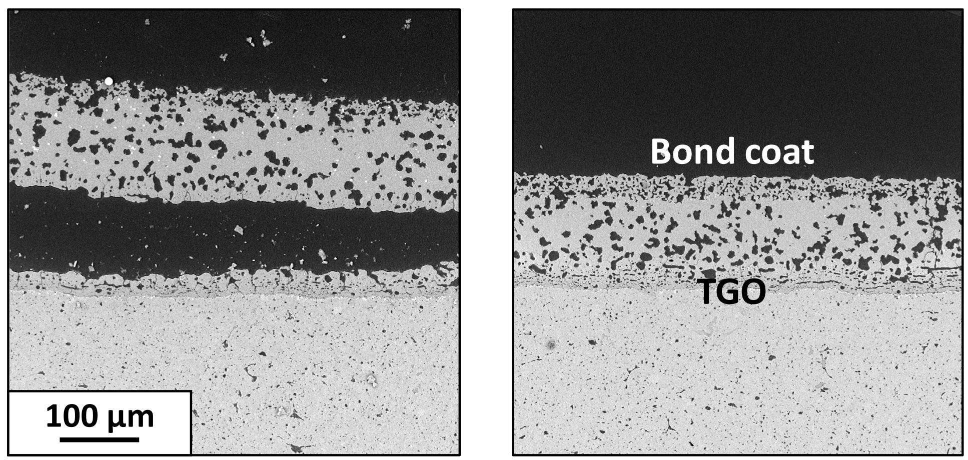
A lower viscosity slurry results in somewhat improved bond coat performance.

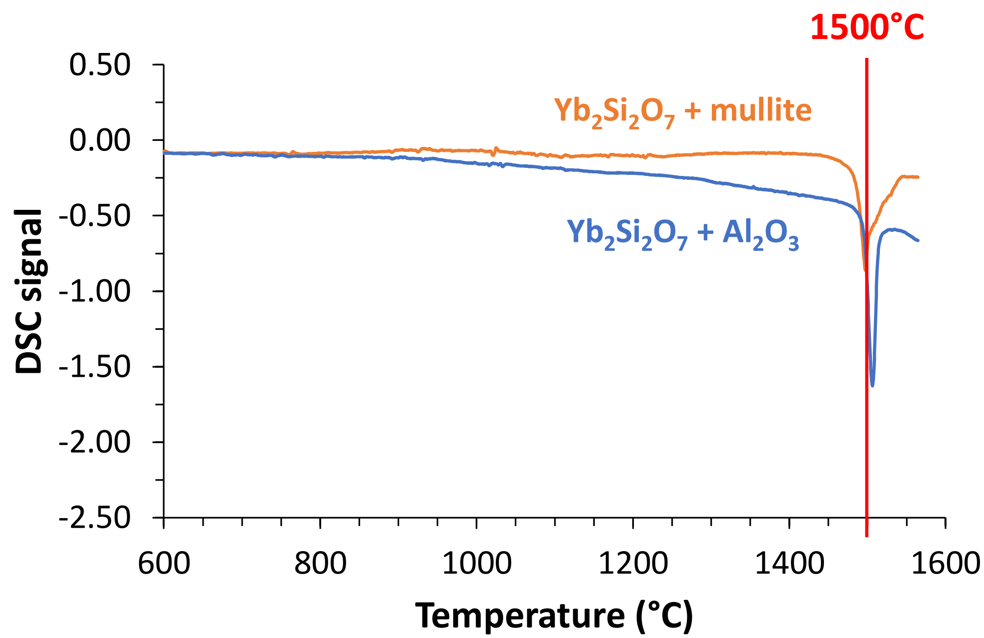
Standard viscosity **Low viscosity**



After 500 cycles, ~1480°C

Results: RE inclusion

- Substitution of Sc or Lu for Yb in $RE_2Si_2O_7$ expected to increase the $RE_2Si_2O_7$ + mullite/Al_2O_3 eutectic temperature



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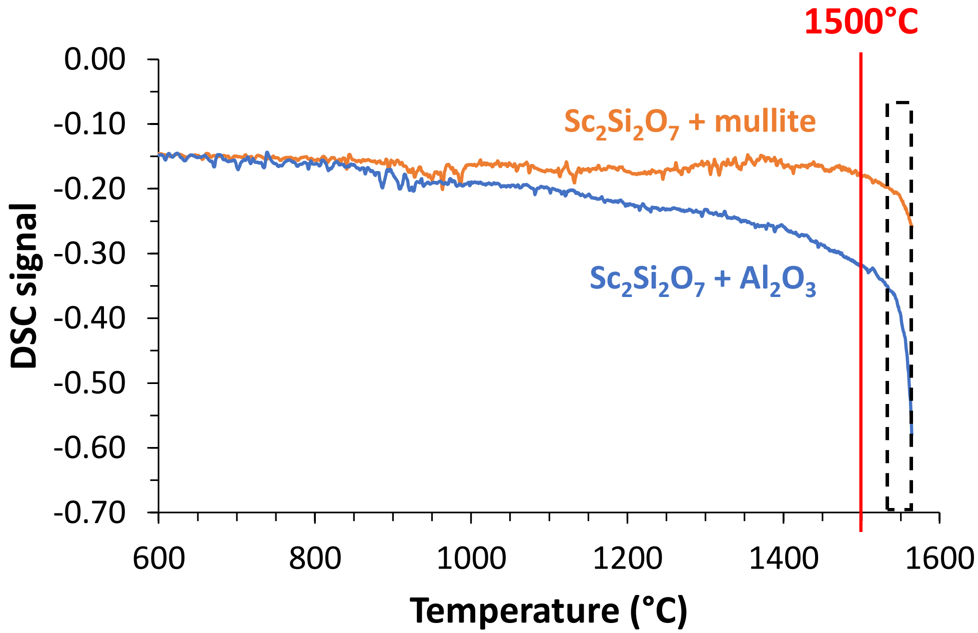

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	Mullite	$RE_2Si_2O_7$	Al_2O_3	Si	SiC
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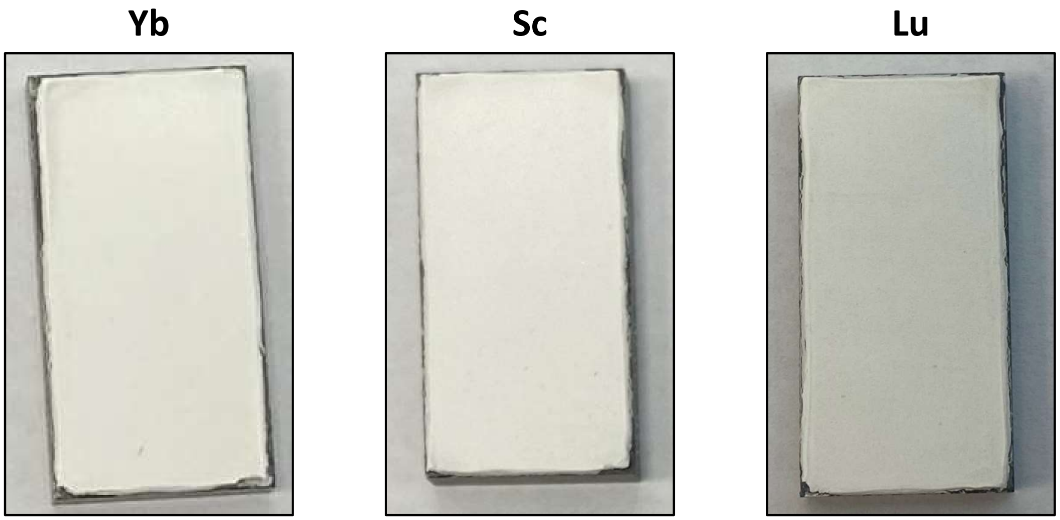
Results: RE inclusion

- 1:1 substitution of Sc or Lu for Yb results in improved bond coat performance
  - Slurry prepared as “low viscosity”; 5 mm milling media



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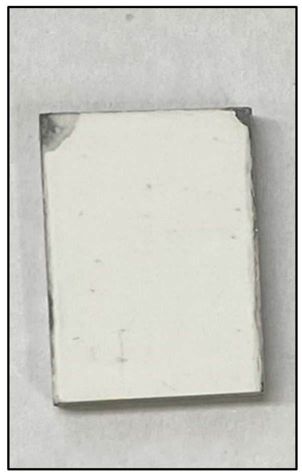
Yb

Sc



Lu

After 500 cycles, ~1480°C

Results: RE inclusion

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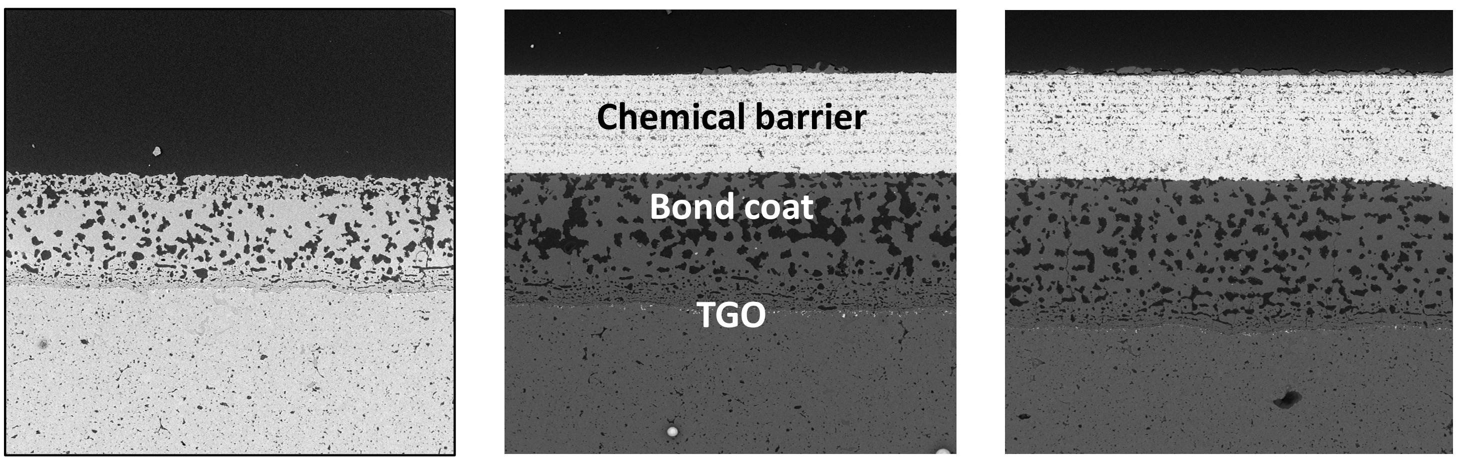
  - Slurry prepared as “low viscosity”; 5 mm milling media

Yb **Sc** **Lu**



After 500 cycles, ~1480°C

Results: RE inclusion

- 1:1 substitution of Sc or Lu for Yb results in improved bond coat performance
  - Slurry prepared as "low viscosity"; 5 mm milling media
- Additional research is underway to optimize the substitution amount
  - Density $Yb_2Si_2O_7$ = 6.15 g/cm^3
  - Density $Lu_2Si_2O_7$ = 6.249 g/cm^3
  - Density $Sc_2Si_2O_7$ = 3.396 g/cm^3

Substitution of the RE in $RE_2Si_2O_7$ has the most pronounced effect on oxide bond coat performance.

Summary

- The effects of particle size, slurry viscosity, and RE chemistry on the performance of a slurry-based oxide bond coat in a steam cycling environment were explored

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Summary

- The effects of particle size, slurry viscosity, and RE chemistry on the performance of a slurry-based oxide bond coat in a steam cycling environment were explored
- There was no trend in bond coat performance as a function of particle size observed over the particle size range investigated
- Lowering the slurry viscosity reduced edge-lifting after processing
- The type of RE in $RE_2Si_2O_7$ had the strongest effect on bond coat performance

Future work

- The effects of particle size, slurry viscosity, and RE chemistry on the performance of a slurry-based oxide bond coat in a steam cycling environment were explored
- There was no trend in bond coat performance as a function of particle size observed over the particle size range investigated
- Lowering the slurry viscosity reduced edge-lifting after processing
- The type of RE in $RE_2Si_2O_7$ had the strongest effect on bond coat performance

- Future work will explore particle size and viscosity effects after bond coat chemistry has been optimized, specifically with respect to RE inclusion
- A slurry-based, HfO_2-dominant top coat chemistry will be investigated